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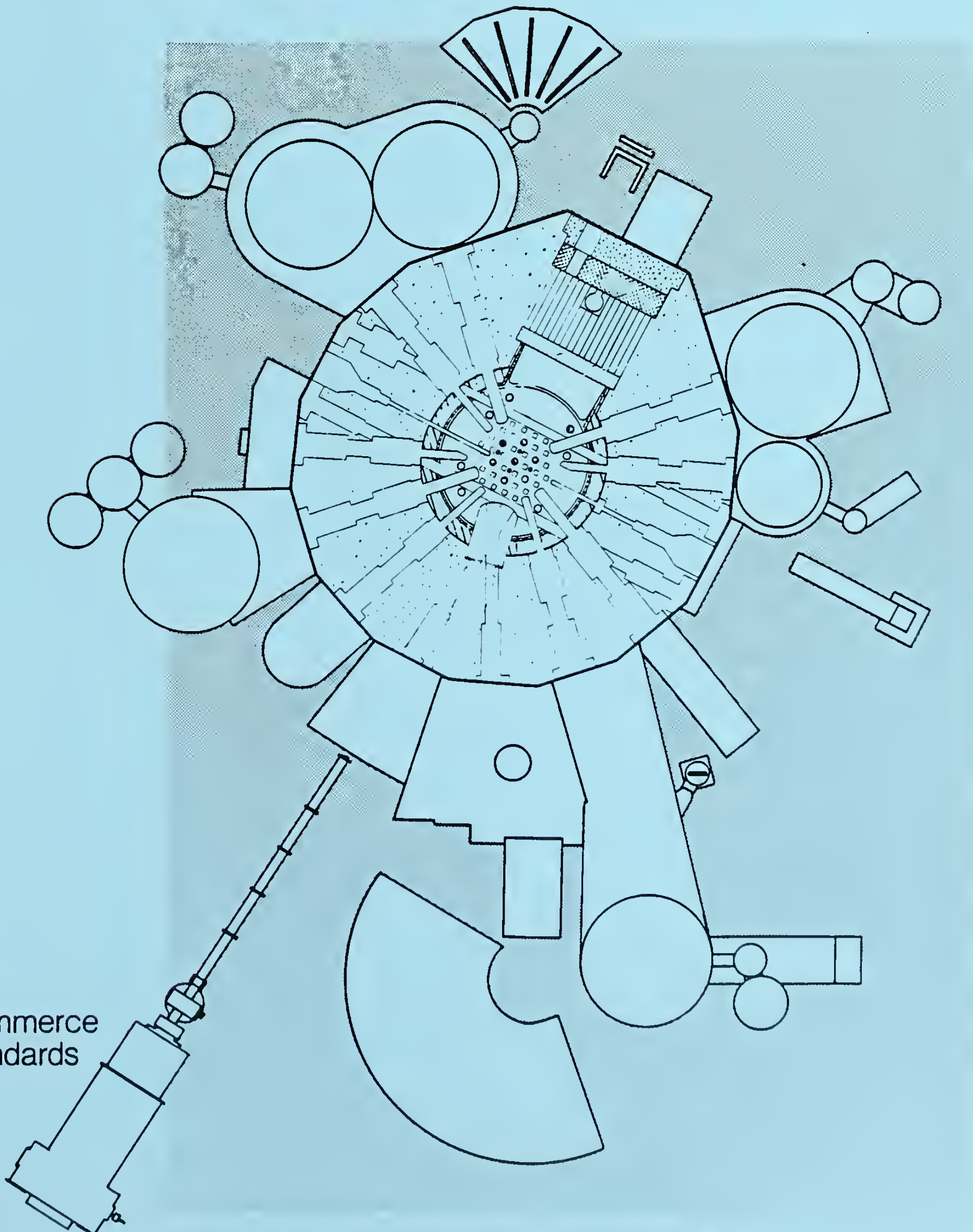
REACTOR RADIATION



NAS-NRC
Assessment Panel
January 20-21, 1987

NBSIR 86-3439
U.S. Department of Commerce
National Bureau of Standards

Technical Activities
1986

QC
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.U56
86-3439
1986

Layout of the NBS reactor experimental facilities and the experimental instruments located on the first floor of the reactor building.

NBS
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1986

IMSE

Institute for Materials Science and Engineering

REACTOR RADIATION

R.S. Carter, Chief
T.M. Raby, Deputy

NAS-NRC
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ABSTRACT

This report summarizes all those programs which depend on the NBS reactor. It covers the period from October 1, 1985 through September 30, 1986. The programs include the application of neutron methods to the characterization of materials, neutron standards, trace analysis by neutron activation analysis, neutron depth profiling, nondestructive evaluation, and the production of radioisotopes.

Key words: Activation analysis; crystal structure; diffraction; isotopes; molecular dynamics; neutron; neutron radiography; nondestructive evaluation; nuclear reactor; radiation.

DISCLAIMER

Certain trade names and company products are identified in order to adequately specify the experimental procedure. In no case does such identification imply recommendation or endorsement by the National Bureau of Standards, nor does it imply that the products are necessarily the best available for the purpose.

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INTRODUCTION AND OVERVIEW

REACTOR RADIATION DIVISION (460)

Robert S. Carter, Chief
Tawfik M. Raby, Deputy Chief
S. E. Tassey, Secretary

The National Bureau of Standards reactor (NBSR) is a national center for the application of reactor radiation to a variety of problems of national concern. The major areas of activity are:

Materials Characterization
Nondestructive Evaluation
Trace Analysis
Radiation Standards and Measurement

The Reactor Radiation Division (RRD), in collaboration with other scientists within the Institute for Materials Science and Engineering (IMSE), other NBS Centers, and outside organizations uses neutron scattering methods to determine the properties and behavior of materials at the submicroscopic level. These methods are used to study a wide variety of problems in such areas as hydrogen in metals, the microstructure of ceramics and metals, microscopic properties of advanced crystalline and amorphous magnetic materials and polymers, molecular species, interactions and pore structures in catalysts and microporous materials, and the structure of biological molecules. These measurements contribute to an understanding of hydrogen effects on metal properties, properties of new and improved alloys and magnetic materials, ceramics and fast-ion conductors, polymer blends, chemical catalysis, and biological processes. NBS is in an excellent position to carry out such a multidisciplinary program because of its strong materials programs located in IMSE and other centers and its 25 state-of-the-art reactor facilities (10 IMSE facilities) that are not available in private-sector laboratories.

Major Activities:

The major research activities of the Reactor Radiation Division involve developing state-of-the-art neutron diffraction, inelastic scattering and radiographic methods and associated experimental facilities, and fostering their application by other NBS divisions and offices and other U.S. industrial, university and government groups to meet critical research needs in physics, chemistry, materials science, and biology. The scientific core group maintains essential research capabilities in condensed-matter science and engages in cooperative research with 200 scientists from NBS and outside groups, including studies of new high technology magnetic and amorphous materials, modern electronic and structural ceramics, chemical catalysts and advanced metals and alloys being developed for new products and technological applications.

In the area of nondestructive evaluation (NDE), RRD uses neutron radiography and both large and small-angle neutron diffraction to examine objects for defects or hidden components that must be examined nondestructively. The major effort is in the development of new or improved neutron NDE methods. Methods are being developed for such diverse applications as the use of autoradiography to study rare paintings and the use of neutron scattering to investigate voids and other defects causing failure in structural materials. This program is partially funded through the Office of Nondestructive Evaluation and is part of the Bureau-wide effort to improve productivity through better quality assurance and increased component lifetime.

Neutron activation analysis is a very sensitive technique for measuring trace elements at very low concentrations. RRD provides the sample irradiations, but the program and sample analysis is carried out by the Center for Analytical Chemistry (CAC). It is used extensively for characterizing Standard Reference Materials (SRM) and a variety of other measurements such as the determination of Iodine-129 concentrations and environmental studies. A large number of outside organizations also use this technique for measuring trace elements or pollutants in foods and drugs, environmental samples, criminal artifacts, etc. This is one of the largest neutron activation analysis activities in the country with thousands of samples irradiated each year. Although the primary effort in this activity is centered around neutron activation analysis, other neutron analytical methods are also being developed. A facility has been built to analyze for trace elements by measuring the prompt gamma ray spectrum induced by neutron capture in the sample, and a facility has been developed (depth profiling) to measure concentrations of light elements (e.g., B, Li) as a function of depth with ~ 100 Å depth resolution, which is the best in the United States.

A program in radiation standards and measurements is carried out by the Center for Radiation Research (CRR). Through the use of double fission chambers and a series of accurately calibrated fission foils, they provide the basis for reactor neutron flux and power density measurements needed in the U.S. fast-flux development program. The calibration and intercomparison of the series of fission foils makes use of standard reference neutron fields established in the thermal column of the NBSR. CRR also maintains well characterized, filtered neutron beams in the reactor of 2 keV, 25 keV, and 144 keV energy for the calibration and development of personnel neutron dosimeters.

A number of other groups both within and outside NBS utilize the long-term irradiation facilities at the NBSR for activities ranging from γ-ray and x-ray physics and standards to application of isotopes in medical diagnosis.

Items of Special Interest

The NBS Reactor has operated very successfully at 20 MW since April 3, 1985. The total operating time since then was 10248 hours, which reflects a 75 % availability, a figure comparable to the best achieved at 10 MW. All experiments at the reactor have realized the expected increase in intensity, and experimenter radiation exposures have not increased significantly from those observed at 10 MW operating power.

The installation of a large-volume cold neutron source in the NBSR in order to increase the flux of long wavelength (cold) neutrons is a high priority of NBS and the Department of Commerce. The first cold source cryostat was received and extensively tested (outside the reactor) in the past year. As a result of these tests, the design was modified in order to increase the flow of He cooling gas through the heavy ice chamber, and the cryostats were returned to the contractor for modification. The first modified cryostat is scheduled for delivery to NBS in November (as of September 25, 1986, the contractor was on schedule), at which time it will again be tested extensively outside the reactor. Assuming that no problems arise during these tests, the cryostat and cold source will be installed in the reactor this winter.

The two prototype instruments being developed with the funding provided by Congress are proceeding well. The new neutron time-of-flight spectrometer has been installed at the CTW beam port (which will be served by the cold source), and the operating parameters are being verified at this time. The first measurements of intensity and resolution are in reasonable agreement with predictions, and the instrument is in the final stages of tuning for operation. This facility will be used at this beam port until the cold source is installed, at which time the intensity will be substantially increased by the enhanced flux of long wavelength neutrons provided by the source. It will be the best instrument in the U. S. A. for studies of quasielastic scattering, allowing entirely new types of research to be carried out, e.g. in the study of the motion of molecules on catalyst surfaces. A spare detector for the Small Angle Neutron Scattering (SANS) instrument has been ordered, and is scheduled for delivery in the coming year. A new data acquisition system capable of handling the increased data rates that will be available with the installation of the cold source is under development. The design is based on the operational system developed for the TOF spectrometer, which in turn is based on the use of a 32 bit minicomputer and a CAMAC interface with histogramming. This system provides a large memory for the storage of data accumulated in real time measurements, and provides a much larger capacity for on-line data reduction. In addition, both instruments are on an Ethernet Local Area Network that connects all instruments in the reactor, and allows remote operation and monitoring of experiments. A high resolution graphics station dedicated to these two instruments is also on this network, as is access to the existing color graphics system.

There have been a number of notable accomplishments in our neutron scattering research programs over the past year, including the collaboration of Purdue and NBS scientists to achieve the first direct observation of charge density waves in a simple metal, using high resolution, high sensitivity crystal spectrometry; and the first neutron scattering study of a new class of multilayer materials composed of magnetic and non-magnetic layers, which has revealed that helical magnetic order in the magnetic layers is propagated through the "dead" layers without loss of phase coherence. In our research on hydrogen in metals, neutron group scientists have achieved the first measurements ever of the phonon spectra of a tritiated material, superconducting $PdT_{0.7}$. These results show that the theory of superconductivity must be reexamined, as it applies to such materials with large mean-square displacements. Another notable accomplishment, in collaboration with Metallurgy Division and U. Illinois scientists, is the first measurement of the neutron diffraction patterns and phonon density of states spectra of a "quasicrystal." These results provide critical information for tests of models of the structure and interatomic forces of this new kind of material discovered at NBS. In the area of instrumentation development group scientists have successfully tested a new graphite/beryllium filter analyzer for neutron spectroscopy, which greatly increases the resolution for studies of the dynamics of catalysts, hydrogen in metals, polymers and other materials, while maintaining very high sensitivity. We have also begun assembly and testing of a new state-of-the-art polarized neutron beam spectrometer, jointly funded by NSF, U.of Md., and NBS.

Finally during FY 86, joint neutron research efforts with industry continued to increase, including e.g.; new or expanded efforts with Exxon, Mobil, and GTE on research ranging from wetting phenomena in microporous media, to molecular processes and arrangements in catalysts, to studies of residual stresses and interatomic structures in new ceramics for automobile and electronic applications.

Reactor Utilization:

As is indicated above, RRD has a dual function. It conducts research programs in the areas of materials characterization and NDE and serves as a focal point of neutron scattering expertise for many other programs both within and outside of NBS. The second function includes not only the operation of the reactor, but also providing sample irradiation services for a large number of users.

An important part of the overall Reactor Division contribution to the NBS mission and to the scientific and technical community is in fostering the utilization of the reactor by other NBS groups and outside organizations. Interactions with other scientists and organizations take the form of both collaborative efforts and independent programs which rely on utilization of the reactor and facilities provided by the NBSR staff. The extent of such interactions for FY 86 are indicated in the tables below. The number of personnel shown in Tables 1 and 2 include many short-term collaborators as well as permanent other agency and university guest workers. These numbers are constantly changing and so may not be exact.

Collaborative interactions are those in which workers from outside the RRD collaborate scientifically with RRD scientists on problems of mutual interest. These interactions are summarized in Table 1.

Many of the other agency and university collaborators have worked with us regularly for many years. Others come from all over the world to spend a few days, weeks, or months to carry out specific experiments using the facilities available at the NBSR. Collaborative programs include measurements on fast-ion conductors, polymers, catalytic materials, hydrogen embrittlement, voids and precipitates in alloys, and ceramics, etc.

Table 1. Collaborative Interactions

	No. of Personnel FY 86
RRD Permanent Scientists	14
Non-RRD Participants	
Other NBS	35
Other Agency	23
University	68
Industrial	35
International	25
Total Non-RRD	186

Independent programs are those programs carried out independently of the Reactor Radiation Division scientific staff by other NBS Divisions and outside organizations. Table 2 summarizes these interactions.

Table 2. Independent Programs

	No. of Personnel FY 86
Other NBS	31
Other Agencies	32
Universities	36
Industrial	25
International	6
Total	130

These tables demonstrate the extensive utilization of the NBS reactor by scientists and engineers from outside the Division. They come from 17 NBS Divisions and offices, 14 other agency organizations, 34 universities, 24 industrial laboratories, and 15 foreign laboratories.

Organization of Technical Activities:

The technical activities of the Division are organized into major tasks. These are summarized below and described in more detail in the "Description of Technical Activities" section. Because the reactor serves not only the Reactor Radiation Division and IMSE, but many other NBS Centers and outside organizations, a description of the major outside activities (Independent Programs) will also be included.

REACTOR OPERATIONS AND SERVICES. Operates and maintains the NBSR, handles all licensing interactions, reactor security, and provides sample irradiation services to a large variety of users. Services range from pneumatic tube irradiations for neutron activation analysis to the production of radioisotopes for medical research. All the many steps necessary to upgrade the NBSR to 20 MW were accomplished under this task.

NEUTRON SCATTERING CHARACTERIZATION OF MATERIALS FOR ADVANCED TECHNOLOGIES.

This task develops and applies neutron inelastic scattering methods and related theoretical analysis for research on the fundamental properties of materials which affect their use in technological applications. Current areas of emphasis include new magnetic and glassy materials, hydrogen in metals, catalytic materials, and graphite intercalation compounds. Task scientists also utilized the special role of neutrons in the structural analysis and nondestructive testing of materials. Task members have competences at the forefront of measurement and analysis of materials structure, including concentrated efforts in neutron diffraction, profile refinement, and state-of-the-art computer methods for: (1) precise structure analysis for effective use of materials (e.g., in electronics, chemical catalysis), (2) development and transfer of an evaluated materials structure database, (3) nondestructive reference methods for texture, and strains affecting materials product processing and performance. The members of this task (and the following task) are also responsible for establishing and maintaining a national center of excellence for neutron scattering, including a computer-controlled network of nine spectrometers at the reactor.

ADVANCED NEUTRON METHODS.

The objective of this competence building task is to develop and maintain new state-of-the-art facilities and technologies for neutron scattering and to further their application in solid-state-physics, chemistry, materials science, and biology. Under this initiative the NBS small angle neutron scattering (SANS) facility, and an improved high resolution crystal spectrometer have been developed and are being widely used for research on the microstructure and dynamics of materials by many NBS divisions, industrial labs and universities. Currently under development is a new world-class ultra-high resolution powder diffractometer for materials structure analysis.

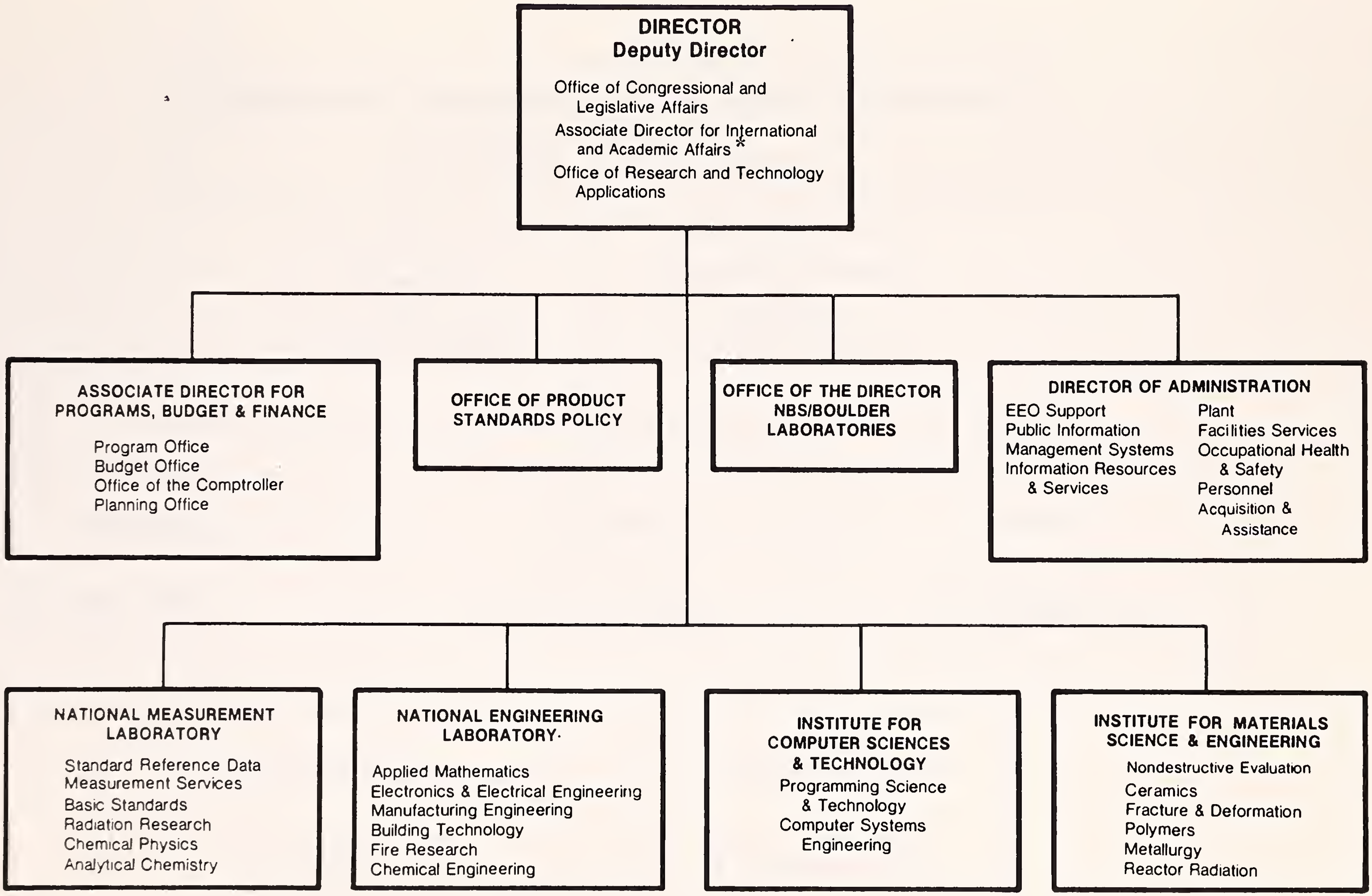
COLD NEUTRON PROGRAM.

The goal of this program is to develop, install and operate a cold neutron source in the NBS reactor, to develop two prototype instruments to utilize the two existing beams from the cold source facility, to conduct scientific research using these two instruments, and to perform preliminary R & D on a large cold neutron research facility that would fully utilize the cold source.

INDEPENDENT PROGRAMS. Although a great deal of research by non-Division scientists is carried out in collaboration with Division scientists, there are many research projects that utilize the reactor and its services, but which are designed and carried out without any scientific collaboration with the Reactor Radiation Division. These Independent programs typically include, trace analysis, depth profiling, radiation standards, neutron dosimetry, environmental studies, and medical research.

ORGANIZATION CHARTS
AND
RESEARCH STAFF

U.S. DEPARTMENT OF COMMERCE
National Bureau of Standards



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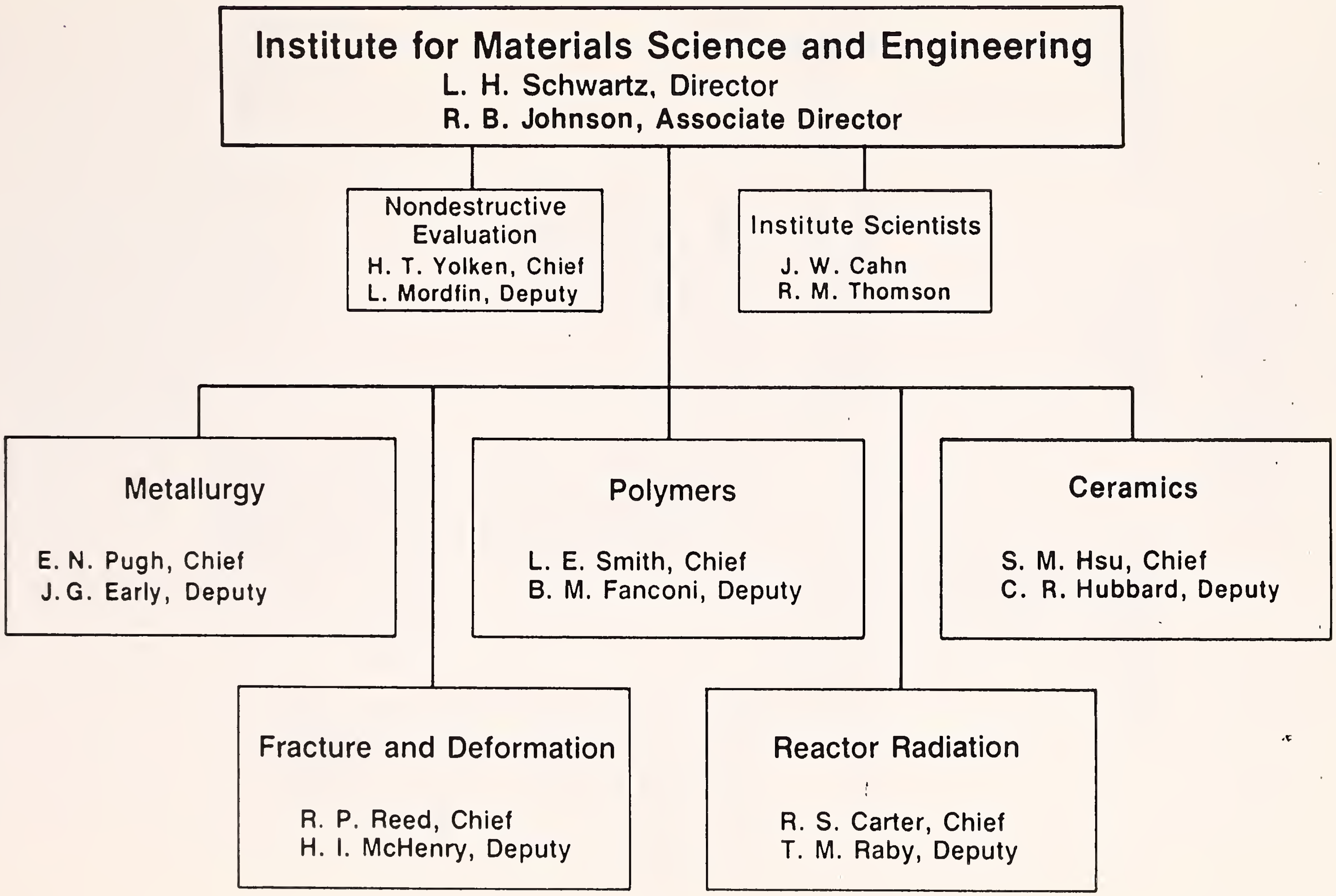
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Ceramics
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Fracture and Deformation
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Reactor Radiation
R. S. Carter, Chief
T. M. Raby, Deputy

REACTOR RADIATION DIVISION

460

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T. M. Raby, Deputy Chief
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*S. E. Tassey, Secretary
S. C. Shatzer, Clerk-Typist

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#T. Cheng

Division Staff

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B. Mozer
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#I. Anderson
C. Glinka (3/4)
+M. Kahn
P. Kopetka
I. Schroder
T. Udovic
R. Williams
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#H. Stanley

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J. F. Torrence, Deputy Chief
C. Harrison, Secretary

Reactor Operators

R. Beasley
M. Bell
N. Bickford
J. Clark
F. Clark
H. Dilks
L. Lindstrom
M. McDonald
W. Mueller
T. Myers
J. Ring
R. Sprow
R. Stiber
A. Toth
D. Wilkison

ENGINEERING SERVICES

J. H. Nicklas, Chief
C. Harrison, Secretary

Mechanical Design

E. Guglielmo
J. Sturrock

Electronics

R. Conway
R. Hayes
L. Shuman

NEUTRON CONDENSED MATTER SCIENCE

J. J. Rush, Chief
M. Clark, Secretary
+L. Clutter, Secretary

Crystallography

E. Prince
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#C. Choi
#V. Himes
J. LaRock (1/2)
A. Mighell
W. Rymes (1/2)
#J. Soubeyroux
*J. Stalick
#F. Beech

Metal Hydrides and Molecular Materials

D. Fravel (1/2)
#H. Prask
J. Rowe (1/4)
W. Rymes (1/2)
#H. Savage
#S. Trevino
#J. Nicol

Magnetic and Amorphous Materials

J. Rhyne
#J. Fernandez-Baca
°R. Erwin
#T. Giebultowicz
*J. Lynn
#P. Mangin
#M. Spano
W. Knill (1/2)
#Y. Yu

Advanced Neutron Methods

N. Berk
D. Fravel (1/2)
C. Glinka (1/4)
J. Gotaas
#S. Krueger
J. LaRock (1/2)

*Part-time
+WAE, Coop, Intermittent
#Guest Scientist, research assoc. (Full Time)
°Post-doc

Research Staff

N. F. Berk	o Condensed matter theory o SANS theory for microstructure analysis o Computer software for graphics and data analysis
R. S. Carter	o Cold Source development o Reactor physics and nuclear engineering
R. C. Casella	o Theory of neutron scattering from light-atom defects in metals o Group theory analyses of neutron scattering from condensed matter o Elementary particle theory, especially as related to reactor generated experiments
C. J. Glinka	o Magnetic materials o SANS microstructure of metals and porous media o Cold neutron instrument development
J. Gotaas	o Low temperature phase transformation o SANS microstructure studies o Magnetism
P. A. Kopetka	o Mechanical engineering o Cold Source design o Electro-mechanical systems
J. LaRock	o Mechanical engineering o Neutron instrumentation design
B. Mozer	o Structure and microstructure of metallic glasses o Dynamics of liquids o NDE of alloys
J. H. Nicklas	o Mechanical engineering o Reactor fuel design o Reactor engineering support
E. Prince	o Structural properties of alloys, catalysts and minerals o Advanced crystallographic refinement methods o Software for materials structure analyses
T. M. Raby	o Reactor operations o Sample irradiations o Reactor standards
J. J. Rhyne	o Properties and transformations of high technology magnetic materials o Structure of amorphous solids o Data acquisition and analysis system

J. M. Rowe	o Orientationally disordered solids o Hydrogen in metals o Cold neutron research and instrumentation
J. J. Rush	o Hydrogen in metals o Catalysts and molecular materials o Two dimensional system
A. Santoro	o Structure of electronic and structured ceramics o Theory of crystal lattices o Powder diffraction methods
I. G. Schroder	o Cold neutron instrumentation development o Nuclear and engineering physics o Optical devices for neutron transport
J. F. Torrence	o Reactor supervision o Reactor maintenance
T. J. Udovic	o Neutron time-of-flight instrumentation o Properties of catalysts o Spectroscopy of surfaces

DESCRIPTION OF TECHNICAL ACTIVITIES

The technical activities of the Division are summarized in this section. A more detailed description of each project can be found in the NBS Technical Note "NBS Reactor, Summary of Activities July 1985 through June 1986."

REACTOR OPERATIONS AND SERVICES

The NBSR is a national center for the application of neutron methods and standards to problems of national importance. The reactor provides intense neutron beams and sample irradiation facilities for more than 300 users from many NBS divisions and outside organizations.

FY 86 Representative Accomplishments

- o More than 135,000 MWH of reactor operation, the best in the history of the facility, were made available to researchers and other users.
- o Development of heavier loaded fuel element that will significantly increase core life and hold down overall fuel costs.

Reactor Operations

T. M. Raby and J. F. Torrence

The past year was the first full year of operation at the higher power level of 20 MW. The new power level proved very successful. Reactor operation was continuous except for periods of refueling and maintenance. During this period, new and refined operating parameters were established that will assure smooth and reliable operation at the higher power level.

Management and shipping of spent fuel is a high priority activity of this task. In the past year, the capacity of the spent fuel storage pool was enlarged to accomodate the increase in spent fuel that has resulted from 20 MW operation. In addition, extensive planning for spent fuel shipment has taken place. In particular, a shipping cask suitable for NBS fuel is under review by the Nuclear Regulatory Commission, and licensing approval is expected later this year. Quality assurance, operating and handling procedures for the use of this cask have been prepared and approved.

A new rule issued by the Nuclear Regulatory Commission requires all research reactors to convert from high enriched uranium fuel to low enriched uranium fuel unless exempted by the Commission. This would have a significant impact on most of the programs at the NBSR because of the resultant reduction in the thermal flux and the decrease in the signal-to-noise ratio. NBS will apply for an exemption from the rule on the basis that important research programs would be compromised by the conversion to low enriched uranium.

A new on-line thermal shield monitoring system along with additional controls have been developed and installed, which resulted in significantly improved performance.

Attracting and keeping qualified operations personnel is becoming exceeding difficult. One experienced reactor supervisor retired, and another experienced senior operator resigned to accept a position in nuclear power. After much effort, two replacements were found. They will require one year of comprehensive training before they become eligible for their licensing examination.

Irradiation Services

N. A. Bickford and J. H. Ring

Extensive irradiation services were provided to many users from within and outside NBS. In all, more than 2,000 irradiations involving thousands of samples were performed.

The Food and Drug Administration continued work at the NBS reactor on improving methodology for determining trace elements in foods and biological materials. Multielement analytical procedures developed at the reactor during the past ten years have been applied to many food types. Work is also being performed on methodology for determining aluminum in biological materials at the 1 ug/g level. The high neutron flux at the NBS reactor makes the facility ideally suited for this program.

A wide range of neutron activation analysis (NAA) procedures have been applied to the determination of trace elements in foods. They include instrumental NAA, pre-irradiation separations, post-irradiation radiochemistry and epithermal NAA. During the past year, detection capabilities were improved for more than ten elements, thus maintaining the role of FDA's NAA facility as a state-of-the-art NAA laboratory. This laboratory facility has been involved in many regulatory investigations, many of which are of major national concern. Included among these was the recent tampering with over-the-counter drugs and packaged foods.

The use of the reactor by the Federal Bureau of Investigation was the heaviest ever, with almost daily activation of physical evidence specimens. The work involved analysis of materials such as gun shot residue and trace elements in hair and metals important in the prosecution of criminal activity. The National Institutes of Health continues to receive fluorine-18 and other radioisotopes produced at the reactor for important medical research.

The University of Maryland continued to make extensive use of instrumental neutron activation analysis (INAA) and neutron-capture prompt gamma-ray activation analysis (PGAA) for advanced development and testing of the methods themselves and to determine concentrations of about 40 elements in particles, gases, rain and fog from the atmosphere, in related source materials such as crystal dust and coal fly ash, and in standards appropriate for use in this field. By measuring concentrations of such a large number of species, it is possible to identify contributions from certain types of air-pollution sources to the atmospheric burden at ambient sampling sites, a technique now known as "receptor modeling." These wide ranging programs involved analyses and studies of samples from natural and man-made sources worldwide, and represent a major step in determining the origin and distribution of pollutants in the environment.

Characterization of irradiation facilities including all the pneumatic tubes and the long-term vertical thimbles was completed. Measurements of flux levels, flux gradients, and thermal to fast flux ratios were made. Similarly, a variety of temperature and pressure measurements for typical samples were also determined. These measurements will provide experimenters with characteristic values for each facility for neutron activation analysis, as well as provide optimum safe operating conditions for use of these facilities.

A variety of other programs used the irradiation services provided by the reactor on a smaller scale.

Engineering Services

J. H. Nicklas and R. S. Conway

In addition to normal engineering and design services provided to reactor operations, experimenters and users, the engineering staff is involved in a continuing effort to upgrade the reactor instrumentation, in addition to normal surveillance calibrations.

The shim arm test assembly used for testing shim arm mechanisms and clutch release assemblies is about finished and is also available for spare parts for reactor shim arms.

Fifty fuel assemblies have been inspected and accepted from the DOE contractor by the NBSR staff. An additional 25 fuel assemblies have been fabricated and are awaiting acceptance. Fuel compacts now being made contain 350 gms per element, a change from 300 gm elements. The first 200 plates of the 350 gm elements will be radiographed to determine that the process is working well.

The fuel element handling tool heads have been modified slightly by adding another spring pin to the mounting block with bars on the lifting shaft to increase its resistance to rotation. This resistance will assure locking of the element to the upper grid and provide operators with a more positive feel when transferring fuel. A new tool for the transfer of fuel from the reactor core to the storage pool was made and tested.

A method has been developed to plug heat exchanger tubes with rubber plugs. The previous method of pounding metal plugs into the tubes required entry into the bonnet with subsequent high radiation exposure to personnel and was limited to personnel that could enter a 12 inch manhole.

A new tool for pulling the fuel transfer arm bearings which will facilitate fuel transfer has been designed and fabricated.

The second tritium monitor has been installed, tested and completed. Two more modern design differential transmitters have been installed in the process demineralized water system.

The instrument group has also provided assistance to the experimental researchers with electronics repair, modification and interface designing. An uninterruptible power supply system was planned, purchased and installation is underway at this time. The UPS system will provide continuous clean power to the experimental computer and associated instrumentation during brief power interruptions.

An instrumentation panel for monitoring the Cryogenics Bismuth Tip experiment facility is in the assembly and testing stage.

NEUTRON SCATTERING CHARACTERIZATION OF MATERIALS FOR ADVANCED TECHNOLOGIES

This task develops and applies neutron scattering techniques, unavailable in the private sector, for precise measurement and research on the structure and microscopic properties of industrial and high technology materials which underlie their processing and use in technological applications. It establishes and maintains a national center and state-of-the-art research facilities using neutron techniques. It also fosters the application of neutron methods to NBS programs and to serve the diverse needs of the U.S. industrial and scientific communities.

Neutron beams are a powerful probe of the critical microscopic properties of materials used in the design, development and production of industrial products, in particular new materials for advanced technologies. The neutron scattering expertise and state-of-the-art, ten-spectrometer network at the NBS reactor are a central resource, not available in the private sector, for 13 NBS divisions and for over 50 U.S. industries and universities (200 participants) which need neutron techniques to address an increasing number of problems and opportunities in materials science, physics, and chemistry. Also under development is a large cold-neutron source and related experimental facilities, which are essential to make materials-based U.S. science and industry competitive with other industrial nations (Western Europe and Japan) in the crucial area of cold-neutron research. Neutron scattering provides critical information on all classes of materials, which cannot be obtained by other techniques. Neutron diffraction and radiographic methods and a materials structure database must be developed and maintained to meet NBS and U.S. needs for more precise structural analysis of materials. Improved capabilities will also be developed to nondestructively measure stresses and defects which affect the performance and failure of modern structural materials. Inelastic, quasielastic, and magnetic scattering methods are tailored as key probes of the microscopic properties of high-technology magnetic materials, industrial catalysts, and superconductors. To serve the crucial U.S. need for cold neutron measurement technology, a large cold-neutron source is being developed to be used for neutron spectroscopy and small angle scattering.

NBS scientists are currently engaged in cooperative research with industrial labs and universities on new or improved materials for semiconductor circuits, power transformers, automobiles and aircraft, sonar and microwave devices, microbatteries and fuel cells, high-strength polymers and ceramics, and chemical catalysts. Neutron methods are essential to many high priority NBS program areas, including surface science, biotechnology, ceramics processing, and nondestructive evaluation. Outside interactions and cooperative materials research underway includes industrial labs (e.g. Exxon, Allied, E-Kodak, IBM, AT&T, W. R. Grace, GE, GTE, DuPont, Mobil), government (NIH, Army and Navy Labs, Sandia, Smithsonian), universities (e.g. U. MD., U. Illinois, U. Calif., Carnegie Mellon, Auburn, Iowa State, Purdue, MIT). Joint research agreements or collaboration also in place with international centers: Institute Laue-Langevin, Saclay research centre, and CNRS (France), Max Planck Institute and KFA, Julich (W. Germany), U. Antwerp (Belgium), Chalmers Institute (Sweden). The materials structure database serves a nationwide user group from industry, universities and government, and has joint agreement with data centers around the world.

FY 86 Representative Accomplishments

- Reactor Radiation Division and U. Illinois scientists have completed the first neutron scattering study of the magnetic order in an epitaxial multilayer of magnetic and non-magnetic rare-earth metals. The neutron results have revealed that the helical magnetic order in the magnetic (Dy) layers is propagated through the intervening yttrium layer and into the next Dy layer without loss of phase coherence, a remarkable result which will significantly influence the tailoring of this novel new class of layered materials for specific magnetic applications.

- Very high resolution diffraction experiments performed over the past several years at the NBS research reactor by scientists from Purdue, U. Missouri, and NBS have produced the first direct evidence for the existence of charge density waves in a simple metal, potassium. Weak satellite diffraction peaks observed in these experiments agree with theoretical prediction that electron density varies sinusoidally with a characteristic wavelength in even the simplest solids. These results provide fundamental new insights into the complicated ways in which electrons behave in materials.

- NBS and Exxon Research and Engineering Company of Annandale, NJ, have signed an agreement that establishes a Participating Research Team (PRT) to develop and operate an improved Small Angle Neutron Scattering (SANS) spectrometer using the cold source at the NBS reactor. The new state-of-the-art instrument, which will be designed and developed jointly by scientists and engineers from the Reactor Radiation Division (IMSE) and Exxon, is expected to be the best in the country, and will be widely used by U.S. industrial and university scientists for a broad range of research in materials science, physics, chemistry, and biology.

- Recent NBS measurements of phonon dispersion curves in $PdT_{0.6}$ (the first ever achieved for a tritiated material) have clearly demonstrated that anharmonicity <u>cannot</u> explain the origin of the novel reverse isotope effect in superconducting PdH. These results show that the theory of superconductivity must be reexamined as it applies to such materials with large mean-square atomic displacements.

- NBS scientists, in collaboration with researchers from the University of Illinois, have performed the first measurement of the vibrational density of states in a "quasicrystal." These neutron scattering results provide a direct test for models of the interatomic forces and dynamics of quasicrystalline materials, which were discovered at NBS three years ago.

- A Memorandum of Agreement has been signed with the Smithsonian Institution to perform neutron autoradiography studies of works of art. The collaborative project will involve the construction of a large shielded enclosure that will make it possible to autoradiograph large paintings at the reactor thermal column. The autoradiograph produces an x-ray like image of the painting revealing any paintings that may be under the surface. The information obtained from these measurements are of great interest to art historians, conservators, art collectors, and galleries, and can often provide important clues relating to the authenticity of the painting.

- Using neutron diffraction, the existence of a long range magnetic ordering has been discovered in bulk dilute rare-earth alloys in a matrix of the non-magnetic metal yttrium. This magnetic behavior has been confirmed in alloys with magnetic concentrations under two percent and has the form of a helix in which the magnetic spins rotate within the material. These results have significant implications for the occurrence of magnetic ordering in new classes of dilute alloys based on spin density waves.

- Joint research efforts with industry in the development and application of neutron scattering methods in catalytic materials have been expanded to include the study of submicroscopic behavior of aromatic molecules in production catalysts with Mobil Research Labs. The Reactor Radiation Divison and the Center for Chemical Physics will cooperate with Mobil scientists in high resolution neutron diffraction and spectroscopic studies of the details of temperature-dependent molecular arrangements and dynamical processes in these materials.

Microscopic Properties of Hydrogen in Metals and Molecular Materials

R. C. Casella, J. M. Rowe, J. J. Rush, W. Rymes, S. F. Trevino,[1] J. Nichol[2]

[1]Guest Scientist, Army Armament Munitions and Chemical Command

[2]Guest Scientist, University of Maryland

Hydrogen in Metals

The NBS neutron scattering facilities continue to be the most sensitive in the world for studying the submicroscopic behavior of hydrogen in metals. Research accomplishments range from the first measurements of phonons in metal tritide crystal to the discovery of an highly unusual potential associated with hydrogen pairing in α-phase yttrium hydride, to the first application of neutron spectroscopy to study clustering of hydrogen around dislocations in a metal.

A primary accomplishment of the past year was the successful measurement of the phonon dispersion curves of palladium tritide ($PdT_{0.7}$). Palladium hydride has been a system of intense interest for many years, most recently since the discovery of high temperature superconductivity (T_c~10K) with a strong inverse isotope effect (T_c(H,D,T)~ 8, 10, 12° respectively). It is also the first known superconductor for which the optical vibration modes provide the dominant election-phonon interaction. These measurements represent one of the most difficult neutron scattering experiments ever performed (because of the small 0.4 cc sample due to radiation safety requirements, and the small tritium cross section). The results demonstrate conclusively that anharmonicity alone cannot, as previously suggested, explain the reverse isotope effect, and that the theory of superconductivity must be reexamined as it applies to materials with the large mean-square displacements characteristic of PdH, PdD and PdT.

Group scientists have also completed a high resolution inelastic scattering study of hydrogen isotopes in hexagonal yttrium metal, which has revealed a highly unusual bonding potential considerably softer and anharmonic along the c-direction than in the basal plane. Low temperature spectra also show a splitting of the c-axis vibrational modes which confirms the pairing of hydrogen on either side of the Y atoms. As another example the first measurements in a study of hydrogen clustering around dislocations in a metal (cold-worked Pd) by neutron vibrational spectroscopy and SANS have been completed. This is the first application of neutron spectroscopy to such a problem. The results thus far have ruled out the models involving "precipitated" hydrogen at the dislocations in favor of a more subtle segregation of H atoms without appreciable metal atom displacements. Preliminary SANS measurements indicate the presence of H "clusters" (density fluctuations) ~ 100 Å in dimension. These results demonstrate the potential use of neutron spectroscopy and SANS in a variety of similar applications correlating hydrogen impurities with other microstructure features affecting the mechanical properties and stability of metals.

Disordered and Catalytic Materials

In our research on catalysts with the Surface Science Division, we have made significant progress in applying neutron spectroscopy to several important catalyst systems. For example, in collaboration with the University of Maryland, we have completed a systematic study of the adsorption and absorption of hydrogen on palladium black, including surface coverage effects. Species associated with surface sites and bulk sites have been clearly identified by differential inelastic scattering spectroscopy, and a vibration mode associated with subsurface H has also been suggested. The character of the surface species has also been studied by a controlled chemical titration experiment, allowing oxygen to attack the surface hydrogen. In another study with Auburn University scientists, we have probed the hydrogen bonding states associated with hydrodesufurization selectivity over RuS_2 catalysts. The vibration spectra show that the surface species are qualitatively independent of adsorption temperature and pretreatment conditions and are primarily sulfhydryl (SH) type groups. Work has also continued on the characterization of molecular species in zeolites by high sensitivity neutron spectroscopy.

As another example of research on disordered materials, group scientists, in collaboration with U. of ILL, have completed the first measurement of the vibrational density of states of a quasicrystal (icosahedral phase) Al_4Mn. Contrary to some predictions, the density of states do not show the existence of localized states and demonstrate that the quasicrystal and crystalline materials are elastically similar, but that the quasicrystal shows a relative excess of phonon density at high vibrational energies. These neutron scattering results provide a direct test for models of the interatomic forces and dynamics of the icosahedral phase. In another area of research with Illinois and Michigan State scientists, we have studied by neutron diffraction and inelastic scattering the structure and phase transformations of new metal-ammonia complexes formed between the layers of graphite. The results thus far indicate that the NH_3 and alkali metals (K) are arranged in closely packed five-member clusters, which undergo at least one ordering transition as the temperature is lowered. Preliminary inelastic scattering measurements have revealed a strong interaction between rotational motions and the two-dimensional translational phonon modes and have also probed the reorientations of both the entire metal-ammonium complex and individual NH_3 molecules.

Microscopic Properties of Magnetic and Amorphous Materials

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[4]Guest Scientists

This subtask provides a focus for the development and application of neutron elastic and inelastic scattering techniques for fundamental studies of the microscopic magnetic properties of new classes of materials (including layered magnetic systems, rare earth metal compounds, magnetic semiconductors, magnetic superconductors, amorphous magnetic alloys and spin glasses), many of which exhibit novel properties of potential use in device applications and high-technology products. This research is carried out in collaboration with many industrial labs and universities.

During the year a number of areas have received intense effort including: (1) the characterization of long-range ordered magnetism in rare earth artificial superlattices, (2) the effects of random exchange on the magnetic states in highly concentrated amorphous iron alloys, (3) the temperature and wave-vector dependence of the spin wave linewidth in amorphous INVAR systems of FeB and FeNiZr, (4) the occurrence of field-induced linear spin density wave states in dilute alloys of Y and Gd, (5) the initial development of a state-of-the-art polarized beam inelastic spectrometer, (6) the damping of magnetic excitations in amorphous FeCr alloys, and (7) the low temperature spin dynamics and pair interaction energies in dilute magnetic semiconductors.

Highlights during the year included:

o Established the existence and character of long range magnetic order in artificial metallic superlattices (multilayers) of magnetically concentrated rare earth elements interleaved with magnetically "dead" yttrium metal layers. These result have significant impact on the theory of magnetic interactions via the formation of spin-density waves in magnetic systems.

o Established the character of the reentrant ferromagnetic to spin glass magnetic state in amorphous alloys of $(FeCr)_{75}P_{15}C_{10}$ using a combination of inelastic scattering and SANS techniques. The spin wave stiffness parameters were observe to soften at low temperatures in contrast to the normal behavior for a ferromagnet, and the lifetimes of the excitations became anomalously short. Concurrent with these effects an elastic scattering component appeared and increased strongly as the temperature decreased.

- Examined the effect of competing exchange interactions present in highly concentrated Fe alloys of FeB and FeZrNi via a study of the temperature dependence of the excitation linewidths. These results conclusively established a fourth power dependence of the linewidth on the wave vector transfer in contrast to the previous reported second power dependence using less accurate methods.

- Demonstrated that neutron scattering can be used to obtain the isolated pair exchange constant in very dilute magnetic semiconductors and thus provided the first reliable data on the magnitude of the nearest neighbor exchange constant in a series of ZnMnS, ZnMnSe, and ZnMnTe magnetic semiconducting systems.

- Studied the inelastic scattering spectra in a unique isotopic sample of CdMnTe which is a magnetic semiconductor. The spectra were found to be in remarkably good agreement with new first-principal calculations of the magnetic excitations in dilute magnetic semiconductors with predominantly nearest neighbor exchange.

- Established the existence of a transition from a helical spin density wave to a linear spin density wave magnetic order on application of an external magnetic field above a critical value in alloys of Gd and Y. The alloys studied were in the dilute limit (1.5 to 4% of the magnetic constituent Gd) and had been previously shown by our neutron studies to have long range magnetic order. The discovery of the field induced transition and its character provides important input to the interpretation of heat capacity and magnetic data and to theoretical model calculations which could not be obtained by normal bulk techniques.

- Examined the nature of the spin excitations in alloys of the rare earth terbium and scandium. These alloys have been shown to be unique in that a "percolation threshold" exists for the occurrence of long-range magnetic order in contrast to other rare earth systems (e.g. Gd-Y, see above) Initial results on the excitation spectra also showed unusual anomalies in the spin wave energies and linewidths as a function of temperature and wave vector transfer.

- Confirmed the lack of a ferromagnetic component to the magnetic order in the reentrant superconductor $Tm_2Fe_3Si_5$ as a function of pressure to 5.8 Kbar. Antiferromagnetic interactions or dynamical fluctuations thus appear to be the mechanism for the destruction of superconductivity in this compound.

- Initiated development of a new polarized beam inelastic scattering spectrometer jointly funded by NBS and the NSF through the University of Maryland. This will be a site-of-the-art machine with many features not present in previous polarized beam spectrometers in this country. It will provide a unique measurement capability for the study of new magnetic materials and hydrogenous solids.

Neutron Diffraction Methods and Application

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Scientists in this group continue to carry out a wide range of crystallographic research, including method development and applications in materials science. This work involves strong collaborative interactions with the Metallurgy, Ceramics, and Fracture and Deformation divisions and with the Center for Chemical Physics in areas as diverse as studies of advanced ceramics for electronic or structural applications, tailored inorganic catalysts, aerospace alloys and quasicrystals, and for NDE studies of residual stress and texture in metals and ceramics. These research efforts often involve joint projects with industries and universities.

As part of our continuing efforts in the study of key structural arrangements related to the tailoring of new or improved catalysts, we have, e.g., completed with DuPont and U. California collaborators a study of the effects of lithium and silver substitution for cesium in the double rings which provide the entry and exit channels for zeolite-rho. While lithium insertion had relatively little effect on ring geometry, silver substitution resulted in major distortion of the framework, which, in fact, is the most distorted ever observed. This work provides valuable insights into the cation exchange mechanisms which can dramatically alter the details of molecular adsorption and release, and thus the specific activity of the catalyst. Work has also continued with Alfred U. scientists on studies of hydroxyl bonds and aluminum deficiencies in activated aluminas which are of high technological importance as catalytic substrates.

As part of our extensive research with the Ceramics Division, AT&T, GTE, and U. of Grenoble, U. of London, etc., on new kinds of ceramics with improved dielectric, or conducting properties, we have completed a detailed study with AT&T of the structural arrangements induced by different heat treatments in perovskite--like $BaBi_{0.5}^{3+}Bi_{0.5}^{5+}O_3$. These neutron diffraction studies have produced critical information on the distribution of Bi ions, which is important for the understanding and tailoring of superconductivity in this class of compounds. As another example, group scientists are collaborating with the Ceramics Division in a systematic study of the alkali aluminosilicates to clarify many important questions related to the crystal chemistry and properties of these materials, including the dependence of coordination cage geometries on alkali metal size, the possible ordering of Al and Si ions, and the subtle role of water molecules in the framework structure.

There have also been a number of new results in our development and application of neutron diffraction techniques for bulk and depth dependent studies of residual stress and texture. For example, residual stress measurements employing energy dispersive techniques have been completed jointly with Army scientists on two distinct alloy types, $UTi_{.0075}$ and 7075-T6 aluminum. The results show clear differences in residual stress patterns related to different fabrication techniques, which have significance for potential stress corrosion cracking problems in aluminum components and uranium alloy fabrication processes. These methods have also been extended with GTE scientists to initiate studies of residual stress in ceramic engine components. In addition, group scientists have joined with the Fracture and Deformation Division for new systematic studies of bulk texture profiles in mechanically deformed copper and steels.

Finally, in the area of condensed matter physics, the neutron group in collaboration with Purdue, have utilized the very high resolution, low background capabilities of the BT-4 spectrometer to directly confirm the existence of charge-density waves in a simple metal, potassium. This result is of great interest and impact in the theory of electrons in metals. Other work has included the first neutron diffraction study of the structure of quasicrystals, with the Metallurgy Division, and cooperation with the Center for Chemical Engineering in a new effort in the study of the structure of organic fluids and mixtures under shear.

NBS Crystal Data Center

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The NBS Crystal Data Center is concerned with the collection, evaluation, and dissemination of data on solid-state materials. The Center maintains a database which includes chemical and crystallographic information on all types of substances with known unit cells. The materials fall into the following categories: inorganics, organics, organometallics, metals, intermetallics, and minerals. For each substance, there are a variety of input and derived data parameters including: the conventional and reduced cell parameters, the space group, the compound name and formula, calculated density, literature reference, and critical comments. The data are used by the industrial and academic communities for a variety of research and analytical purposes including the identification and characterization of materials. The work of the NBS Crystal Data Center is carried out in close collaboration with allied data centers in the United States, England, Canada, and Germany. Some recent accomplishments are summarized below.

A major effort has focused on preparing the next version of the NBS CRYSTAL DATA Distribution Package for official release in 1987. Both the database and software components of this package are being upgraded. The database will be expanded seven times over the present version and will include ~ 115,000 entries with 1.7 million records of data. Each entry will include all data types that are input or created by the NBS*AIDS83 program. An NBS Technical Note (in press) has been written that describes conventions and formats for all data records in the database.

To permit scientists to use the database, we have developed software tools that can be distributed with the database or used to search the database on-line at a central site. NBS software (NBS*LATTICE), which is an integral part of the CRYSTAL DATA Distribution Package, is designed to be used on a variety of computers. With the lattice-matching program function, unknown compounds can be identified by comparison with entries in the database once any cell of the lattice has been determined using diffraction techniques.

A very promising method for data distribution is made possible by access to central on-line systems. Thus using the CISTI-NBS CRYSTAL DATA Search System, the database can be accessed worldwide. All key data items can be searched and answers to queries can be obtained using Boolean type searches. During the year, the system has been greatly expanded with respect to the data (~ 115,000 entries) and available software tools. The system is routinely used by scientists to solve a variety of scientific problems in diverse areas of chemistry, solid-state physics, and materials science.

Radiographic Materials and Standards

R. S. Carter, M. Ganoczy, Y. J. Cheng,[1] and J. S. Olin[1]

[1]Research Associate, Smithsonian Institution

A Memorandum of Agreement was signed this year with the Smithsonian Institution to expand studies of works of art by neutron autoradiography techniques. It is planned that the collaborative project will involve the construction of a large enclosure to make it possible to autoradiograph large paintings at the NBSR. One great strength of neutron radiography is that it stands alone as the only available method that allows the observation of multiple pigment applications beneath the surface of an oil painting. It thus offers art historians and conservators a very special opportunity to observe the working method of an artist, quite literally from the ground up. Great progress was made during the past year, e.g. , in an ongoing systematic study of the works of the prominent turn-of-the century American Impressionist, Thomas Wilmer Dewing. The work so far has provided a number of insights into the techniques of this artist over his career. Each of the five paintings radiographed appear to have been initiated and constructed in a different manner, varying from a line drawing, to several stages of painting over, to a premier coup with no preparation at all. This evidence related to the evolution of technique along with detailed information on pigments and their origin offer an increasingly powerful probe to improve understanding of the art of master painters here and abroad, while providing a valuable evaluation tool for judging the authenticity of works of art.

COLD NEUTRON PROGRAM

The goal of this program is to develop, install and operate a large volume cold neutron source in the NBS Reactor, to develop two prototype instruments to utilize the two existing beams that view this source, to conduct scientific research using these two instruments, and to develop plans for a large experimental hall with cold neutron guides that would fully exploit the potential of the cold neutron source. (Funding of this project began in FY1985, and six new engineering and staff members have been brought on board).

FY 86 Significant Accomplishments

- Testing of the first cryostat for the cold neutron source was completed. All of the test results except for the flow of cooling gas through the system were satisfactory, including the making of ice in the cryostat, achievement of the desired temperatures, vacuum integrity, helium jacket operation and operation of the refrigerator-cryostat assembly. A design modification to increase flow was mocked up and tested, and a contract to modify the cryostats was issued.

- Fabrication of the neutron Time-of-Flight (TOF) spectrometer was successfully completed, and the spectrometer was installed and aligned at the CTW beam port (one of two which will be served by the cold neutron source). Preliminary tests of the intensity and resolution are in satisfactory agreement with expectations.

- A new SANS data acquisition system was purchased, and the interface was designed, fabricated and tested. This system will be installed and commissioned when the cold source is installed.

- The conceptual design of a large cold neutron experimental facility was completed, including a new neutron guide tube layout. The new design allows the same capacity as the previous design, but allows a superior floor design at a substantially reduced cost.

Cold Neutron Source

R. S. Carter, J. M. Rowe, P. A. Kopetka and R. S. Williams

The use of cold neutrons (*i.e.* neutrons with wavelengths and energies characteristic of temperatures $\leq$ 40 K) is important to many areas of materials science, including the study of microstructure in alloys, polymers and polymer blends, and biologically significant molecules; the study of the motions of large molecules and molecular assemblies; the use of depth profiling to study sub-surface impurities and dopants; and the study of the structure and dynamics of novel materials. The normal reactor moderator produces a spectrum in which the cold neutrons are only 1 % of the total number. However, by replacing a portion of the normal moderator with a cryogenically cooled moderator, this fraction can be increased substantially. The NBSR was designed with a large volume suitable for the insertion of a cold neutron source, and we are installing a 38 cm. block of D_2O ice cooled to 25 K into this volume. As was reported last year, we received the first of two cryostats for this source, and during the past year a series of out-of reactor tests were completed and analysed. As a result of this analysis, we concluded that the cooling flow through the moderator was too low, and re-designed the cooling coils (which are imbedded in the D_2O ice). We then tested a mockup of the new arrangement (a difficult task, since a true test requires the end product - a cryostat), and having satisfied ourselves that the results were satisfactory, negotiated and awarded a contract for modification of both cryostats. The first cryostat is scheduled for delivery to NBS in November, 1986, and is on schedule at the time of writing (September, 1986). When it is delivered, it will be subjected to another series of tests before insertion into the reactor. The installation date will depend on the tests, but is tentatively scheduled for early winter. However, in view of the nature of the installation, it will not be done until we are completely satisfied with all possible out-of-reactor tests. In the meantime, the instrumentation for the large refrigerator is being upgraded, and a spare compressor is being installed in order to add to redundancy of critical systems.

Instrumentation and Facility Design

J. M. Rowe, T. Udovic, I. G. Schroder, C. J. Glinka, G. Greene, J. Larock, and D. Fravel

When the cold source is operational, it will be viewed by two beam ports, CTW and CTE, at which are installed a Time-of-Flight (TOF) spectrometer and a Small Angle Neutron Scattering (SANS) instrument respectively. These two instruments are the only ones which can be served within the confines of the current reactor confinement building, and we have proposed a new experimental guide hall for construction adjacent to the Reactor building. This facility has been included in the Presidential budget for Fiscal Years 1986 and 1987, but budget constraints have prevented funding of this proposal as of this date (September, 1986). However, we have continued to refine our plans and specifications for this proposed facility, and in the past year we have been able to produce a modified design incorporating a new layout of neutron guides and a new floor design that has reduced the cost significantly without compromising the final capacity of 16-18 experimental stations. Detailed design cannot begin until approval is received from Congress, but we continue work on conceptual design of the building and associated instrumentation.

The TOF spectrometer has been installed at the CTW beam port, and is in the process of final commissioning for general use. The initial tests of intensity and resolution are in satisfactory agreement with predictions, and the data acquisition system, which will be the prototype for both the SANS instrument and any new spectrometers built in association with the guide hall, is essentially complete. When the cold source is installed, this instrument will be unrivaled in the U.S. for the study of the molecular and atomic motions of many systems such as molecules and molecular assemblies on catalysts, hydrogen in metals, and complex molecules in the solid or liquid state or in solution.

The SANS instrument is installed at the CTE beam port, and we continue to upgrade this facility in order to handle the increased capability of the cold source. Among other upgrades underway, such as procurement of a spare area detector, a new data acquisition system, based on the operational TOF system, is being assembled for this facility. This system will be able to accommodate the large amount of data that will be produced by real time studies, and will provide greatly enhanced on-line data reduction capabilities for the users. The software will feature a user-friendly interface based on menu driven operation, and will also allow operation from remote terminals through an Ethernet Local Area Network, with enhanced graphics capabilities.

Research Program

J. M. Rowe, T. Udovic, J. W. Lynn[1], H. Stanley[1], J. Nicol[1], S. Krueger, C. J. Glinka, and J. J. Rush

[1]Guest Scientist, University of Maryland - NBS Cooperative Agreement

The most important aspect of this entire project is to ensure that the cold source and associated instrumentation are properly utilized to produce the best possible science. We have therefore initiated research efforts both within and outside NBS. First results with the Center for Chemical Physics, Dupont and the University of Maryland on the bonding states and reorientational dynamics of ammonium species in zeolites have been completed and new experiments are being planned with Mobil researchers on the molecular arrangements, dynamics and interactions of aromatic hydrocarbons in an industrial catalyst, using high resolution cold neutron spectroscopy and diffraction. A co-operative research arrangement is also underway with Exxon Research and Engineering, with a joint post-doctoral associate for the use of SANS to study a number of materials problems, including melting in micro-porous media. During the coming year, our cold neutron TOF spectrometer will also be critical to joint research on bonding and diffusion mechanisms in hydrogenous ionic conductors, and to the study of low lying excitations in high technology magnetic materials.

ADVANCED NEUTRON METHODS

N. F. Berk, D. Fravel, C. J. Glinka, J. Gotaas, J. LaRock, and E. Prince

This is one of the NBS competence building initiatives whose objective is to develop and maintain state of-the-art facilities for advanced neutron scattering techniques and to further the application of these techniques in solid-state physics, chemistry and materials science. Under this initiative the small angle neutron scattering (SANS) facility for microstructure studies in the 10-2000 A range has been built and continues to be improved. An improved high resolution crystal spectrometer has also been developed for dynamic studies. An important part of this initiative has been the fostering of collaborative programs with a number of NBS divisions, universities, and industrial and government laboratories to develop new science and diverse applications of these advanced neutron techniques.The new project now underway in this task is the development of an advanced instrument for ultra high-resolution powder diffraction which will provide the best measurement capability of its kind in the United States. This facility will be widely used in NBS programs and by U.S. scientists from industries and universities.

FY 86 Representative Accomplishments

- Staff scientists have successfully demonstrated the use of a beryllium-graphite band-pass filter analyzer to greatly increase (by a factor of ~ 3) the resolution available for "incoherent" neutron spectroscopy between 25 and 100 meV, while maintaining high sensitivity for studies of the dynamics of catalysts, hydrogen in metals, polymers, and other molecular materials.

- As part of a collaborative effort with the University of California, the first SANS study of precipitate coarsening in reactor pressure vessel steels involving in-situ specimen annealing has been carried out. A vacuum furnace which mounts between the pole pieces of a high field electromagnet was developed for this purpose. The SANS results and associated microhardness tests are being used to develop relationships between the total fluence and temperature, and the precipitate population and hardness of the steels.

Facilities

1. Small Angle Neutron Scattering (SANS)

While the SANS instrument operated virtually without interruption during the past year, improvements in the areas of sample environmental apparatus and data acquisition proceeded in parallel. For example, a programmable temperature controller and power supply were implemented which can monitor and control the temperature of a variety of vacuum furnaces, as well as the instrument's multi-specimen sample changer. One such furnace, designed and built by the Nuclear and Chemical Engineering Dept. of the University of California at Santa Barbara, fits between the pole pieces of a high field electromagnet to enable samples to be annealed in-situ at temperatures up to 600C in an applied field of up to 18 kilogauss.

An advanced data acquisition system for the SANS instrument based on a Microvax II computer is now under development an will replace the existing system during the reactor shutdown for installation of the cold source. The new system will utilize CAMAC-based histogramming memory to offload the basic data acquisition tasks from the CPU which will then be available for online display and analysis. This system will allow rapid switching among several data storage arrays to enable the response of samples to cyclic perturbations (e.g. stress, shear, fatigue, etc.) to be studied. In addition, the instrument computer has sufficient speed and memory to enable all routine types of data analysis to be carried out while an experiment is in progress. With these improvements the SANS instrument will be optimized to utilize the increased neutron flux produced by the cold source.

2. Crystal Spectrometer

The development of new higher-resolution capabilities for the BT-4 spectrometer has been extended to the development and testing of a new beryllium-graphite band-pass filter-analyzer for neutron spectroscopy. The new analyzer provides an energy window ~ 3 times narrower than a conventional beryllium filter and makes possible for the first time, high sensitivity neutron spectra between 30 and 100 meV with resolutions between 1.7 and 2.5 meV. This new capability has already been used with great success in studies of the anomalous potential experienced by H isotopes in yttrium and in measuring the density of states of quasicrystalline Al_4Mn. This analyzer will have a great impact on neutron studies of the vibrational spectra of catalysts, molecular solids, and many other materials.

3. Ultra High-Resolution Powder Diffraction

Progress this year toward the development at NBS of the world-class ultra-high resolution neutron powder diffractometer included (1) the design and fabrication of a prototype of the 6 minute collimators which are a key to the performance of the instrument; (2) completion of procurement of the 35 neutron detectors and associated electronics for the new multidetector instrument and; (3) successful testing of the first set of copper monochromators which will be used at one of the diffractometer takeoff angles.

Research and Measurement

In FY 86, a record number of scientists (60) from government, university, and industrial laboratories and eight NBS divisions carried out measurements on the SANS instrument. The bulk of this work can be broadly classified under three areas: microstructural studies of metallic, ceramic or catalytic materials, measurements of molecular conformation of polymers and biomaterials, or studies of magnetic correlations and phase transitions in novel magnetic materials. Inelastic scattering with the high-resolution crystal spectrometer involves measurements of wave vector dependent excitations and diffusive processes in materials.

SANS measurements in collaboration with the Center for Analytical Chemistry have proven successful in determining the effective thickness of monomeric and polymeric hydrocarbon chains grafted to the pore surfaces of silica microparticles. The SANS measurements have provided the first direct evidence of the effect of various solvents entrained in the pores on the effective thickness of the adsorbate layers. These results have a direct bearing, for example, on understanding (and tailoring) the performance of chemically modified microparticles in reverse phase liquid chromatography.

The tendency of low concentrations of hydrogen in α-palladium to cluster under the influence of large internal stresses and dislocations, has been convincingly demonstrated in a collaborative effort with the Max Planck Institute involving both SANS and inelastic scattering measurements. Characteristic cluster sizes of about 100Å were observed in mechanically deformed Pd foils while no evidence for clustering was seen in strain-relieved foils, a result with significant implications for studies of hydrogen embrittlement.

A joint NBS-NIH-University of Maryland collaboration in using SANS to characterize the structure and interactions in concentrated protein solutions has been extended to the examination of proteins in red blood cells and neurosecretory vesicles. From measurements at appropriately chosen conditions of scattering contrast, information on the internal organization of a particular type of protein can be obtained under conditions of greatest physiological relevance.

As part of an ongoing collaboration with the University of California at Santa Barbara in using SANS to study microstructural changes related to embrittlement in reactor pressure vessel steels, the first in-situ annealing studies have been carried out of the growth of precipitates rising from impurities in the steels. The presence of radiation damage is known to accelerate the precipitate growth which is now being quantified by SANS and associated microhardness tests. Additional measurements on model alloys prepared from separated isotopes (to control the scattering contrast) are providing new information on the composition of the precipitates.

Through the use of deuterium labeling to highlight the network structure in epoxies, scientists in the Polymer Division have obtained significant results with SANS regarding the molecular modes of deformation in strained epoxies. They have observed in epoxies with short crosslink spacings typical of commercial materials that there is essentially no change in the average crosslink distance in samples strained up to 30%. This remarkable result suggests that the macroscopic deformation is controlled by some type of defect motion analogous to the way dislocations determine strain in metals.

The first proprietary measurements using the SANS facility have been carried out, under recently established NBS guidelines, by polymer scientists at the Eastman Kodak Company and the IBM Almaden Research Laboratories.

INDEPENDENT PROGRAMS

The two major independent (non-collaborative) Bureau programs using the reactor are nuclear methods group in the Center for Analytical Chemistry and standard neutron fields for neutron flux calibration and materials dosimetry in the Center for Radiation Research. These programs will be summarized here. The major non-NBS independent programs were summarized under the irradiation services provided by Reactor Operations.

FY 86 Representative Accomplishments

- A novel analytical procedure has been developed for the measurement of baseline levels of lithium and boron in biological materials with the combination of neutron activation followed by helium gas mass spectrometry. The determination of these elements at their normal levels in body fluids has been a challenge, due in part to the likelihood of contamination for any method requiring dissolution. The nondestructive nature of the new method means that concentrations at the ng/g normal levels can be measured reliably. The quantification of the lithium is based on the helium-3 decay product of the tritium from the Li-6 (n,a)Li-7 reaction. To avoid helium production by fast neutron reactions, all irradiations were done in the thermal column of the NBSR. The mass spectrometry has been carried out at the Physics Department of McMaster University. Extension of the method to metals, ceramics and geological materials is underway.

- The NBS ^{235}U Cavity Fission Source at the NBS Reactor was used this past year to expose U-238 to a certified neutron fluence of $2x10^{16}$ n/cm^2, which is sufficient to produce a measurable 30-year Cs-137 fission-product activity. This is the first time such a standard, which is used, e.g., to verify commercial neutron fluence measurements for reactor pressure vessel surveillance, has ever been produced.

NUCLEAR METHODS

R. Fleming

The development and application of nuclear analytical techniques for greater accuracy, higher sensitivity and better selectivity are the goals of the Nuclear Methods Group. A high level of competence has been developed in reactor-based activation analysis (INAA and RNAA), and in LINAC-based activation analysis using photons (PAA). In addition, the group has a unique activation analysis capability in neutron beam analysis with both prompt gamma activation analysis (PGAA) and neutron depth profiling (NDP). The NDP technique utilizes prompt charged particle emission to determine elemental distributions within the first few micrometers of a surface while the PGAA technique utilizes prompt gamma-ray emission to measure the total amount of an element in a sample, regardless of its distribution. These techniques provide an arsenal of tools to address a wide variety of analytical problems in science and technology.

The activities of the past year have been highlighted by our involvement in the 7th International Conference on Modern Trends in Activation Analysis, new measurements carried out at other research facilities, and by a major renovation of our laboratory space in the Reactor Building. The MTAA-7 meeting, which was held in Copenhagen in June, commemorated the fiftieth anniversary of the discovery of activation analysis by Hevesy and Levi. NBS scientists were authors of ten of the papers presented at this conference. The opportunity was also provided for visits to the laboratories of several of our European colleagues.

In addition to our ongoing use of the nearby reactors at the University of Maryland and the University of Virginia, primarily for fast and epithermal activation analysis, Group members carried out measurements on the neutron beam facilities at the new cold neutron source at Julich and the intense guided thermal neutron beams at Grenoble. The Julich work, which was reported at the MTAA-7, provided the first demonstration of the use of cold neutron beams for prompt gamma activation analysis. At the ILL reactor, which currently has the world's most intense thermal neutron beams, the neutron depth profiling facility was used to demonstrate the profiling of oxygen with the $^{17}O(n,a)^{14}C$ reaction. This work has since been repeated on the NBS facility, but the actual implementation of NDP for oxygen must await the very intense beams of the proposed NBS Cold Neutron Facility to be practical.

The Group's contribution to the certification of Standard Reference Materials is illustrated by the multielement measurements done on the following SRM's: Fly Ash, Flint Clay, Plastic Clay, Dolomitic Limestone, Rice Flour, and Wheat Flour. At major levels, the determination of silicon by fast neutron activation analysis has been demonstrated. Preliminary measurements of trace levels of lithium and boron in biological materials will be published using a thermal neutron irradiation followed by a helium gas mass spectrometric determination for quantification.

Research into biomedical problems has again been strong this year centered around the activities of the joint NBS/NOAA/EPA Environmental Specimen Bank. A new stable isotope technique for the measurement of total blood volume in humans has been reported based on the NAA determination of the Cr-51/Fe-59 ratio in samples spiked with Cr-50. The characterization of biological macromolecules by a combination of gel electrophoresis and neutron activation analysis is under development. A special large volume irradiation facility has been constructed for this purpose. A systematic effort has begun on the accurate and efficient measurement of trace elements in human serum, focused initially on zinc and selenium. The crucial step of valid sampling of biological materials was reviewed in a special issue of the Bureau's Journal of Research.

The strong interaction with industrial scientists using neutron depth profiling, prompt gamma activation analysis, and neutron activation analysis has continued with a large number of guest workers, research associates, and joint publications. The longterm NDP study of the mobility of helium in nickel has been completed. The measurement of distributions of lithium and boron in metals, glasses, and polymers continues to produce important results.

The Joint NBS/FDA/USDA study of trace elements in human diet, sponsored by the International Atomic Energy Agency, has completed its second year. A total of 30 biologically important minor and trace elements have been measured on the total diet material, USDIET-I. A second composite diet material is being prepared for study and as a candidate for an NBS Reference Material.

During the coming year the Group will continue to improve the accuracy and sensitivity with which nuclear methods are applied to elemental measurement. This will address the problems of sample preparation, irradiation, and counting with the goal of reducing and quantifying the various sources of systematic error in analysis by nuclear methods.

Neutron Field Standards and Dosimetry

J. Grundl

The Neutron Dosimetry Group of the Center for Radiation Research (CRR) is engaged in the development and application of standard and reference neutron fields as permanent facilities for neutron detector calibrations, and for reaction rate cross section measurements. Strong interactions with outside organizations, both in the federal and private sector, are important programmatic characteristics. A substantial component of the Group's activities involve the use of the NBS Research Reactor thermal column and tangential beams where facilities designed and built by the Neutron Dosimetry Group are in operation.

Neutron Personnel Dosimetry

In neutron personnel dosimetry the Neutron Dosimetry Group's activities include the use of the NBS standard neutron fields for routine calibrations, for non-routine testing, development, and calibrations of new types of neutron instrumentation, and for quality control measurements of production instruments. A tissue equivalent proportional counter (TEPC) system to determine neutron dose as a function of energy deposited is being developed under contract with the Armed Forces Radiobiology Research Institute (AFRRI), and the first operational tests of the system have been successfully conducted at the AFRRI TRIGA reactor. Also, a new type of neutron dosimeter is under development in cooperation with Yale University and a remmeter based on a TEPC with Battelle Pacific Northwest. New fission chamber monitors for the AFRRI exposure rooms have been fabricated and tested. These monitors will provide a direct measure of neutron exposure over the full range of the TRIGA reactor dose rates, without sensitivity to gamma ray background. Based on a double ionization chamber arrangement, these monitors will establish exposures with a precision of one percent for power levels as low as one kW and irradiations as short as one minute, or alternatively, will monitor exposure rates at a power level of one MW with less than one percent deadtime correction.

Dosimetry for Materials Performance Assessment

Neutron fluence standards are neutron sensors (activation foils generally) in which a radioactive species relevant for dosimetry is induced by irradiation in a standard neutron field. The NBS U-235 Cavity Fission Source, at the NBS reactor, was used to expose U-238 to a certified neutron fluence of about 2 X 10^{16} n/cm^2 which is sufficient to produce a measurable 30-year Cs-137 fission-product activity. This is the first time such a standard has ever been produced. This fluence standard is used to verify commercial neutron fluence measurements made in connection with vessel surveillance. Furthermore, reactor pressure quality assurance measurements of tantalum impurity in niobium were performed for several commercial and government laboratories using the thermal neutron irradiation facility at the reactor. These measurements were made in support of the wide interest in the use of the $^{92}Nb(n,n')^{92m}Nb$ reaction for fast neutron fluence measurements.

Integral Neutron Cross Section Measurements

As a result of a DOE sponsored multi-laboratory, multi-nation effort to measure the fission-spectrum-averaged cross section of the $^{92}Nb(n,n')^{m92}Nb$ reaction for high fluence (> 10^{16} n/cm^2), a 30-day (10^{17} n/cm^2) irradiation has been completed at the NBSR ^{235}U Cavity Fission Source Facility. The 30-day irradiation, an essential component of the measurement, was 6 times longer than any previously performed at this facility.

The collaboration with the University of Virginia Department of Nuclear Engineering and Engineering Physics is continuing. The photo-fission cross sections of Np-237, U-235, U-238, and Th-232 have been measured in the NBS neutron driven (iron and cadmium) gamma-ray fields. A paper will be presented at the 6th ASTM-Euratom Symposium on Reactor Dosimetry. These cross sections are important for correcting the neutron response of fission dosimeters used in all areas of materials performance assessment under stress of radiation. Also, as part of this collaboration the analysis of the data from the $^{92}Nb(n,n')^{m92}Nb$ is being performed at the University of Virginia. Integral tests of 10-100 keV-range neutron activation cross sections for Au, In, and Cu have begun at the Intermediate Energy Standard Neutron Field (ISNF) facility at the reactor thermal column. The ISNF is ideally suited to cross section tests in this 10-100 keV region, and these tests may contribute to understanding the inconsistencies in the B-10 cross section in this energy range.

NBS Cavity Fission Neutron Irradiation Facility

Several man months of effort were spent this year in documenting the calibration services offered in association with the NBS Fission Neutron Irradiation Facility. Four documents entitled Activation Foil Irradiation-Cavity Fission Sources (44090C), now in the NBS review process, will be published as and NBS Internal Report. This document describes the characteristics, theory, and operation of this benchmark neutron field, including the traceability of its calibration to NBS-I. Available neutron fluences are given along with the uncertainty of relevant exposure parameters.

Neutron Life-Time Experiment

Preliminary work in support of a proposed neutron life-time experiment has begun. The responsibility of the Neutron Dosimetry Group in the proposed experiment would be the determination of the average liner density of neutrons in the neutron beam, downstream from an electromagnetic proton trap. A detachable neutron detector would be absolutely calibrated in a series of subsidiary measurements employing both cold neutron beams and thermal neutron beams. The active neutron detector will consist of a thin B-10 deposit, viewed by four surface barrier detectors. A study of possible materials for the backing of the B-10 deposit has been made, and thin

(0.38 mm) crystalline silicon wafers have been chosen as the backing material. These single-crystal backings will scatter thermal neutrons less than half as much as would the thinnest possible Al backing (0.13 mm). Furthermore, these crystal wafers are extremely smooth, flat, and rigid, so that good solid angle reproducibility and stability can be maintained.

OUTPUTS/INTERACTIONS

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Technical and Professional Committee Participation and Leadership

American Crystallographic Association
Crystallographic Computing and Data Committee, A. D. Mighell, Member

American Crystallographic Association
Neutron Scattering Special Interest Group, E. Prince, Chairman

American Crystallographic Association
Small Angle Scattering Special Interest Group, C. Glinka, Secretary

American National Standards Committee
N-17: Research Reactors, Reactor Physics, and Shielding
R. S. Carter, Chairman, T. M. Raby, Secretary

Argonne National Laboratory, Pulse Neutron Sources
Program Review Committee, J. J. Rhyne, Member

American Nuclear Society
ANS-15 Standards for Research Reactors, T. M. Raby, Member
Standards Steering Committee, R. S. Carter, Voting Member

BNL Beam Facility Users Committee, J. J. Rush, Member

DOE Basic Energy Science Advisory Committee, J. M. Rowe, Member

Fe-based Multilayer Session of Magnetism and Magnetic Materials (MMM)
Conference, Baltimore, Md., R. Erwin, Chairman

Gordon Conference on Orientational Disorder in Crystals, 1986
J. M. Rowe, Chairman

International Centre for Diffraction Data
Data Base Subcommittee, J. K. Stalick, Member

International Conference on Magnetism, 1985-1986
Publications Chairman and Member Executive Committee, J. J. Rhyne

International Union of Crystallography, Subcommittee on Statistical
Descriptors, E. Prince, Member

Joint Committee-Crystal Data Task Group, A. D. Mighell, Member

Journal of Applied Crystallography, Journalist Committee of the
International Union of Crystallography, E. Prince, Co-editor

Journal of Applied Physics, 1986 Member, Editorial Board, J. J. Rhyne

National Bureau of Standards and JCPDS--International Centre for Diffraction
Data, Crystal Data Management Board, A. D. Mighell, Member

NSF Review Team for MRS at Michigan State, J. J. Rush, Member

Seminar on International Research Facilities, American Physical Society Representative on Large Matter Condensed Matter Science Facilities, Organizing Committee, J. J. Rush, Member

Steering Committee for an Advanced Steady State Neutron Source in the United States, R. S. Carter, Member

Steering Committee for an Advanced Steady State Neutron Source in the United States, J. J. Rush, Chairman

Workshop on Advanced Steady State Neutron Facility, Organizing Committee, J. J. Rush, Member

Industrial and Academic Interactions

As a national center for the development and application of neutron methods in condensed matter and materials science, chemical analysis and radiation standards, the Reactor Radiation Division, currently has direct interactions and cooperative programs with 34 universities, 24 industrial and 15 foreign laboratories. A few examples of these many interactions are:

- A new collaborative research effort has been initiated with Mobil to apply neutron diffraction and inelastic scattering in probing of molecular arrangements and dynamics which are a key to understanding and improving chemical processing in prototype catalysts.

- An agreement was established between NBS and Exxon Research and Development Corporation to jointly develop and operate a world-class small angle neutron scattering spectrometer at the NBS cold neutron source. An interim cooperative research effort has been established at the existing SANS instrument at the reactor in research on wetting in microporous media and micellar systems.

- The Reactor Radiation Division's Crystal Data Center is engaged in a number of interactive links including joint development and distribution to U.S. science and industry of evaluated crystal data with the International Center for Diffraction Data. The Data Center also has long-term agreements in place with crystal data programs in Canada, Great Britain, and Germany to jointly develop and share critical data on the structure of materials.

- Cooperative Research efforts with the Physics Department and Materials Research Laboratory at the University of Illinois were greatly expanded during the past year to include the first neutron diffraction work worldwide on a new class of layered magnetic materials and the initiation of research on new kinds of metal-molecular complexes created within the layers of oriented graphite.

- Cooperative Research Program with the Department of Physics and Astronomy of the University of Maryland. Under the program RRD staff are engaged with Maryland scientists in joint research on magnetic materials, catalysts, biological materials, and in the development of state-of-the-art polarized neutron scattering instrumentation. Some of this research is carried out jointly with scientists from industrial labs.

- The joint catalytic materials program between Auburn University and the Reactor Radiation Division at the National Bureau of Standards is concerned with utilizing neutron scattering techniques in conjunction with more traditional catalysis research methods to investigate the influence of adsorbate orientation, surface stoichiometry, and crystallite size/morphology on metal sulfide catalyst selectivity toward important hydrotreating applications.

- Collaborative research activities with GTE Research Laboratories have been extended to the application of neutron diffraction and small angle scattering techniques to study the structure, residual stress, and microstructure in new ceramics used for electronic and auto-engine components.

Associated Activities

During the past year, scientists in the Reactor Radiation Division delivered some twenty-seven invited lectures in the U.S. and abroad. The Division also coorganized with the Department of Energy and private sector laboratories a workshop for planning a new advanced steady state neutron facility in the United States. Neutron Group scientists also received European support for cooperative research efforts abroad including a NATO grant for research with West German scientists on hydrogen in metals and a position as visiting scientist at the Chalmers Institute in Sweden for collaborative development of advanced crystallographic methods and instrumentation. Finally, the Division Chief, Robert S. Carter was elected a fellow of the American Nuclear Society this past year.

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10. SUPPLEMENTARY NOTES

☐ Document describes a computer program; SF-185, FIPS Software Summary, is attached.

11. ABSTRACT (*A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here*)

This report summarizes all those programs which depend on the NBS reactor. It covers the period from October 1, 1985 through September 30, 1986. The programs range from the use of neutron beams to study the structure and dynamics of materials through nuclear physics and neutron standards to sample irradiation for activation analysis, isotope production, radiation effects studies, neutron radiography, and nondestructive evaluation.

12. KEY WORDS (*Six to twelve entries; alphabetical order; capitalize only proper names; and separate key words by semicolons*)

Activation analysis; crystal structure; diffraction; isotopes; molecular dynamics; neutron; neutron radiography; nondestructive evaluation; nuclear reactor; radiation.

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